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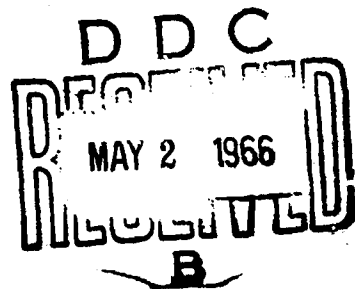
Second Quarterly Progress Report

Composition Research Unit
Chemistry Section
Research Division

Rocketdyne
A Division of North American Aviation

April 1966

Group 4
Downgraded at 3-Year Intervals
Declassified After 12 Years



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Air Force Rocket Propulsion Laboratory
Research and Technology Division
Edwards Air Force Base, California
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FOREWORD

The research reported herein was supported by the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Edwards Air Force Base, California, Air Force Systems Command, United States Air Force, under contract number AF04(611)10809, project number 1148, BPSN 623148, program structure number 750G, with Ralph Fargnoli, 2nd/Lt/USAF, RPCL, serving as Project Monitor. This quarterly technical progress report was prepared under Rocketdyne G.O. 8728 in compliance with Part I, Paragraph B, and line item 6 of DD1423, and IL-STD-847 (USAF).

The work described covers the period 1 October 1965 through 31 March 1966. The Responsible Scientist for this program is Dr. Hubert E. Dubb of the Analytical Chemistry Group, headed by Dr. B. L. Tuffly. The work was carried out by members of the Composition Research Unit supervised by Dr. V. H. Dayan, the Chemical Sensor Research Unit supervised by Mr. I. Lysyj, the Santa Susana Field Laboratory Chemical Analysis Unit supervised by Miss N. V. Sutton, the Measurement Systems Unit supervised by Mr. R. E. Nelson, the Synthetic Chemistry Group headed by Dr. E. A. Lawton, and the Propellant Engineering Group, headed by Dr. J. J. Kalvinskas.

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ABSTRACT

This program was originally concerned with evaluating a new storable liquid oxidizer, INT0, which is NT0 inhibited with a fluorine-containing oxidizer, F_3NO .

Storability results have shown that F_3NO is not compatible with NT0 at elevated temperatures. $FN0_2$ has been substituted as the inhibiting fluorine oxidizer and this new propellant system is called INT0-2. Its constituents have been found to be compatible at 70 C.

Long- and short-term corrosivity tests have been initiated. The 7-day compatibility tests with non-metallics have been completed in NT0, INT0-1, and INT0-2. Results indicate that INT0-2 is more compatible than INT0-1 with the materials studied. There was little difference between NT0 and INT0-2.

Conductance measurements were completed on INT0-1. Results indicated that galvanic corrosion will not be a problem.

INT0-2 has been prepared by reaction of fluorine with NT0 and the resulting mixture has been shown to react with added water to bring the water equivalent down to 0.03 percent.

Analytical methods have been developed for the analysis of INT0-1 and INT0-2.

(Confidential Abstract)

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INTRODUCTION

The use of nitrogen tetroxide (NTO), the most widely used storable liquid oxidizer in the United States, has been continually hampered by corrosion problems. Dry NTO is not a highly corrosive liquid, but moist NTO is extremely corrosive because of the formation of nitric and nitrous acids by the reaction of NTO with water. The present program was undertaken with the objective of determining whether an oxidizer system consisting of NTO with a small amount of F_3NO added would be less corrosive than moist NTO. F_3NO was chosen as an additive because it had been demonstrated by nuclear magnetic resonance (n.m.r.) spectroscopy and by Turner bulb water equivalency measurements that this compound reacted with the nitric and nitrous acids present in moist NTO to form HF and to greatly reduce the nitric and nitrous acid content of the propellant. The other reaction anticipated was the one between O_2 produced by the F_3NO -acid reaction and the NO present in NTO, the product of which was NO_2 . Thus, no new harmful species were formed.

The technical position at the start of the program was then one of knowledge that (1) dry NTO is not highly corrosive, (2) wet NTO is highly corrosive, and (3) adding F_3NO to wet NTO reduces the nitric and nitrous acid content of the propellant with the concurrent production of HF. In addition, n.m.r. spectra taken over a 1-week period indicated no significant decomposition of the F_3NO -NTO system.

As a result of compatibility tests made since the start of the program, it is now known that the F_3NO -NTO system is not storable at elevated (53 and 70 C) temperatures because the constituents react to form FNO_2 . FNO_2 has therefore been substituted for F_3NO as the additive. The compatibility of FNO_2 with NTO has been demonstrated at 53 and 70 C. Also, it has been shown that FNO_2 is as effective as F_3NO in reducing the water content of moist NTO.

As a consequence of the experimental results summarized above, the objective of the program has been changed from evaluating the operational capability of INTO ($F_3NO + NTO$) to evaluating the operational capability

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of INTO-2 ($\text{FNO}_2 + \text{NTO}$). The revised objectives of the program are to make an engineering evaluation of the corrosion properties and storability of INTO-2, to determine some of its basic physical and chemical properties, and to develop methods for its chemical analysis.

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PHASE I: ENGINEERING EVALUATION

PHYSICAL TESTING

Corrosivity Tests

The tests and evaluations applicable to INTO-1 ($\text{NTO} + \text{F}_3\text{NO}$), as defined in the first quarterly report, are also applicable to INTO-2 ($\text{NTO} + \text{FNO}_2$). The list of materials will be repeated because of some minor changes from the original list (Table 1).

TABLE 1

MATERIALS TO BE SUBJECTED TO CORROSIVITY TESTING

Group I Iron Alloys	Group II Aluminum Alloys	Group III Nonmetals
304 Stainless Steel	6061-T6	Kel-F
316 Stainless Steel	7075-T73	Teflon
321 Stainless Steel	Tens 50	Viton A
440C Stainless Steel	2219-T71	Kynar
AM 350 SCT	2014-T6	Phenolic Resin-Cured Butyl Rubber
1018 Carbon Steel	2024	
Weld Samples	Weld Samples	

In the Group I iron alloys, 321 stainless steel was added because it is a representative, weldable, stainless steel and does not require the special annealing which is required by 316 stainless steel to reduce oxidization at the weld.

For the Group II aluminum alloys, the 2024 alloy was substituted for the 2219 alloy which was unavailable at this time but should be available approximately 1 May 1966. Because the 2024 alloy has chemical, physical

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and corrosion resistance properties quite similar to the 2219 alloy, it was substituted in order not to delay the balance of the testing. A comparison of the chemical properties is shown in Table 2. The 2024 and 7075 aluminum alloys are considered "not weldable" because at this time they are only weldable under controlled laboratory conditions and not normally welded in production. As a result, only nonwelded specimens of the 2024 and 7075 alloys will be tested.

Viton A, military specification MIL-R-25897 Class I, has been added to the Group III nonmetals, because of its possible use in NT0 systems as an O-ring material.

Because the investigation is comparative in nature, and its objective is to evaluate the effectiveness of the additive in preventing corrosion, specimens will be exposed to five propellant compositions. These are:

1. NT0 (military specification, 0.10 weight percent H_2O equivalent and a minimum 99.5 percent assay)
2. NT0 + 0.2 weight percent H_2O
3. NT0 (military specification) + 2.0 weight percent $FN O_2$
4. NT0 + 0.2 weight percent H_2O + 2.0 weight percent $FN O_2$
5. NT0 + 0.5 weight percent HF

All specimens were cleaned using a soap solution followed by a water rinse, followed by an acetone rinse. The specimens were then dried and weighed to the nearest 0.10 milligram. All weights were recorded as shown in the sample form presented as Table 3 .

A coding system was established to define each specimen and its test conditions and is delineated in Table 4 . Letters were assigned for each material, numbers were assigned for each test condition, and Roman numerals were assigned for each propellant composition.

The test bombs, valves, and fittings were vapor degreased, rinsed with trichloroethylene and then rinsed with acetone. The valves were completely disassembled for cleaning. Fluorolube was used as a lubricant on the valve stem assemblies.

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TABLE 2

COMPARISON OF CHEMICAL COMPOSITION

Aluminum Alloy	Alloy Elements, percent										
	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	V	Zr	Al
2014	0.5-1.2	1.0	3.9-5.0	0.4-1.2	0.2-0.8	0.1	0.25	0.15	—	—	Remainder
2024	0.5	0.5	3.8-4.9	0.4-1.2	0.05	0.1	0.1	—	—	—	Remainder
2219	0.2	0.3	5.8-6.8	0.2-0.4	0.02	—	0.1	0.02-0.1	0.05-0.15	0.10-0.25	Remainder

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TABLE 3
RECORDING WEIGHTS

MATERIAL: 304 L Stainless Steel, Code Letter A, With Weld Bead					
Code	Starting Weight, grams	Weight After, grams	Weight Change, gm	Comp	Comments and Metalurgical Observations
L I Q U I D P H A S E	A-1	2.5954		I	
	A-5	2.6451		II	
	A-9	2.5663		III	
	A-13	2.8196		IV	
	A-17	2.0594		V	
	A-21	2.8311		I	
	A-25	2.5726		II	
	A-29	2.3024		III	
	A-33	2.8763		IV	
	A-37	2.1428		V	
	A-41	2.1450		I	
	A-45	2.6206		II	
	A-49	2.5269		III	
	A-53	2.8019		IV	
	A-57	2.4270		V	
V A P O R P H A S E	A-3	2.1296		I	
	A-7	2.7431		II	
	A-11	2.7722		III	
	A-15	2.1421		IV	Small metal whisker on specimen on edge
	A-19	2.5568		V	
	A-23	2.1913		I	
	A-27	2.8816		II	
	A-31	2.9798		III	
	A-35	2.3819		IV	
	A-39	2.6932		V	
	A-43	2.4086		I	
	A-47	2.6352		II	
	A-51	2.9236		III	
	A-55	2.3527		IV	
	A-59	2.6285		V	

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TABLE 4

SPECIMEN ALPHANUMERIC CODING SYSTEM

Alphabetical Connotation

<u>Iron Alloys</u>	<u>Code Letter</u>	<u>Aluminum Alloys</u>	<u>Code Letter</u>	<u>Nonmetals</u>	<u>Code Letter</u>
304 Stainless Steel	A	6061-T6	G	Kel-F	N
321 Stainless Steel	B	7075-T73	H	Kynar	O
440 C Stainless Steel	C	Tens-50-T6	I	Butyl rubber	P
AM 350 SCT	D	2219-T71	J	Viton "A"	Q
1018 Carbon Steel	E	2014-T6	K	Teflon	R
316 Stainless Steel	F	2024-T6	L		

Numerical Connotation

<u>Circumstance</u>	<u>Numerical Notation</u>
Liquid Phase, Welded	1, 5, 9, 13, ---57
Liquid Phase, No Weld	2, 6, 10, 14, ---58
Vapor Phase, Welded	3, 7, 11, 15, ---59
Vapor Phase, No Weld	4, 8, 12, 16, ---60
30-Day Test at Ambient Temperature	1, 2, 3, 4, ---20
20-Month Test at Ambient Temperature	21, 22, 23, ---40
30-Day Test at 70 C	41, 42, 43, ---60

Propellant Composition Designation

<u>Composition</u>	<u>Roman Numeral</u>
NT0 (MIL Spec 0.10 weight percent H_2O equivalent and a minimum 99.5 percent assay) ²	I
NT0 + 0.2 Weight Percent H_2O	II
N_2O (MIL Spec) + 2.0 weight percent $FN O_2$	III
NT0 + 0.2 Weight Percent H_2O + 2.0 Weight Percent $FN O_2$	IV
NT0 + 0.5 Weight Percent HF	V

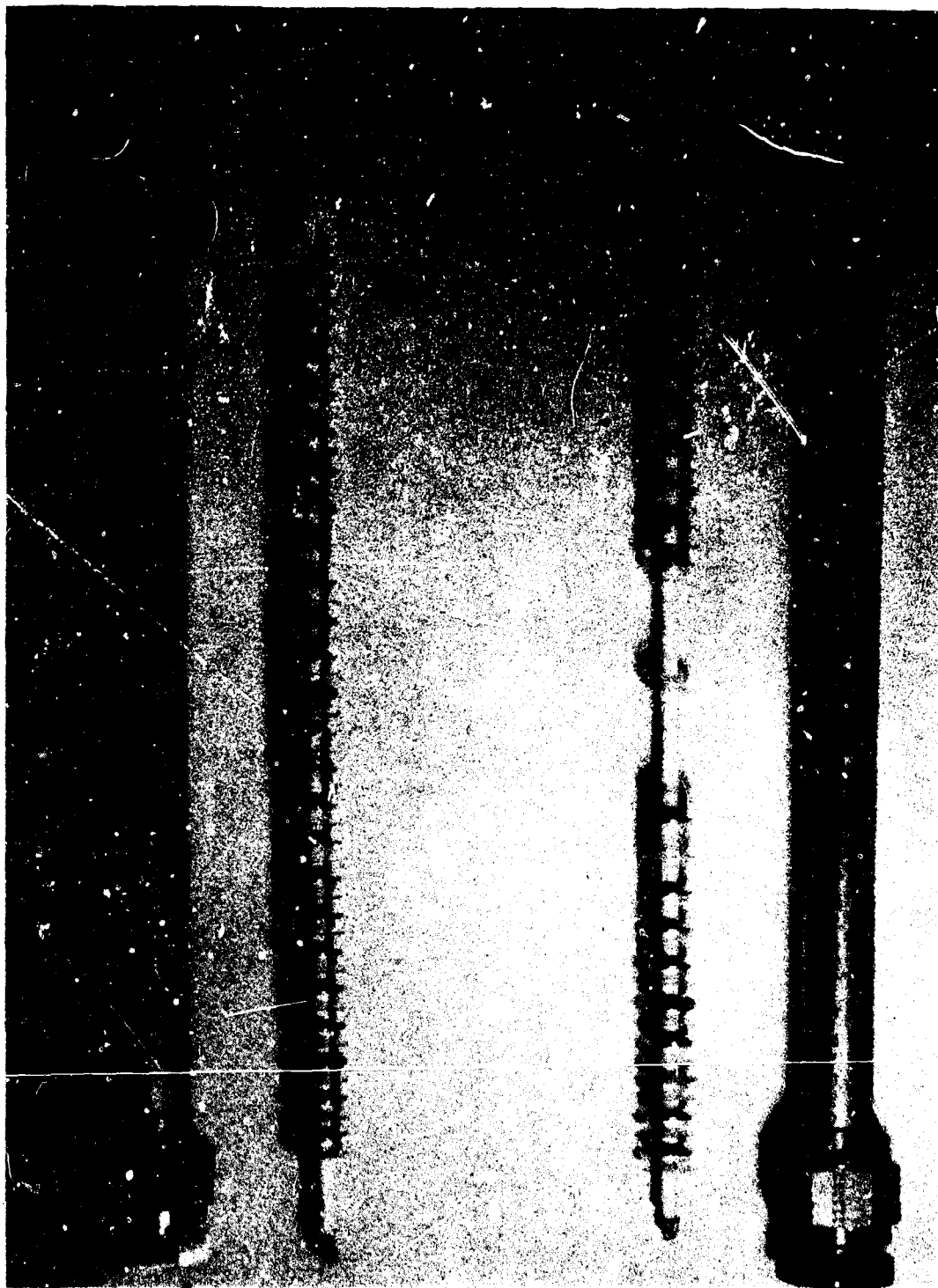
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Test specimens were assembled on rods using Teflon spacers and Kel-F separators between each specimen (Fig. 1). A detailed description was presented in the first quarterly report.

The test bomb is loaded on to a vacuum rack (Fig. 2). The space between valves No. 1 and 2 is evacuated, valve No. 2 is closed, and valve No. 1 is opened. With valves No. 3 through 5 open, the test bomb and loading assembly are evacuated. Valve No. 4 is closed and the loading assembly is pumped down to a high vacuum. Valve No. 3 is closed and valve No. 4 is opened, so that the test bomb is pumped down to a high vacuum. At this time the dewar flask is filled with liquid nitrogen. Valve No. 2 is opened, and the loading assembly is allowed to fill for 5 minutes. After the loading assembly is filled, valves No. 2 and 5 are closed isolating the loading system from the vacuum system and propellant reservoir. Valve No. 3 is then opened and the transfer is carried out for a given period of time (approximately 10 minutes). During the transfer, the test bomb is kept at liquid nitrogen temperature. Upon completion of the transfer, valves No. 3 and 4 are closed and the bomb is removed from the loading system. The bomb is allowed to warm and then is weighted to verify transfer of a 65-milliliter volume. Smaller bombs and a smaller loading assembly are used for the nonmetal samples.

Evaluation of the nonmetals compatibility tests was based on weight change (when feasible) and visual examination for swelling, dissolution, cracking, etc. Seven-day tests of the compatibility of INTO-1 and NT0 with Kel-F, Teflon, Kynar, Viton A, and Butyl rubber have been completed. Identical specimens were immersed in the liquid and in the vapor phase at ambient temperature. The first three materials appeared unaffected except for color changes. The Viton A cracked in both NT0 and INTO-1 and Butyl rubber was completely dissolved in INTO-1 and seemingly unaffected by NT0. The F_3NO in the INTO was not completely destroyed by the reaction with the Butyl rubber. The immersed and vapor phase specimens seemed to be identically affected during both the INTO-1 and NT0 tests.

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Fe Alloys

Al Alloys

Figure 1. 30-Day and 20-Month Test Specimens and Bombs

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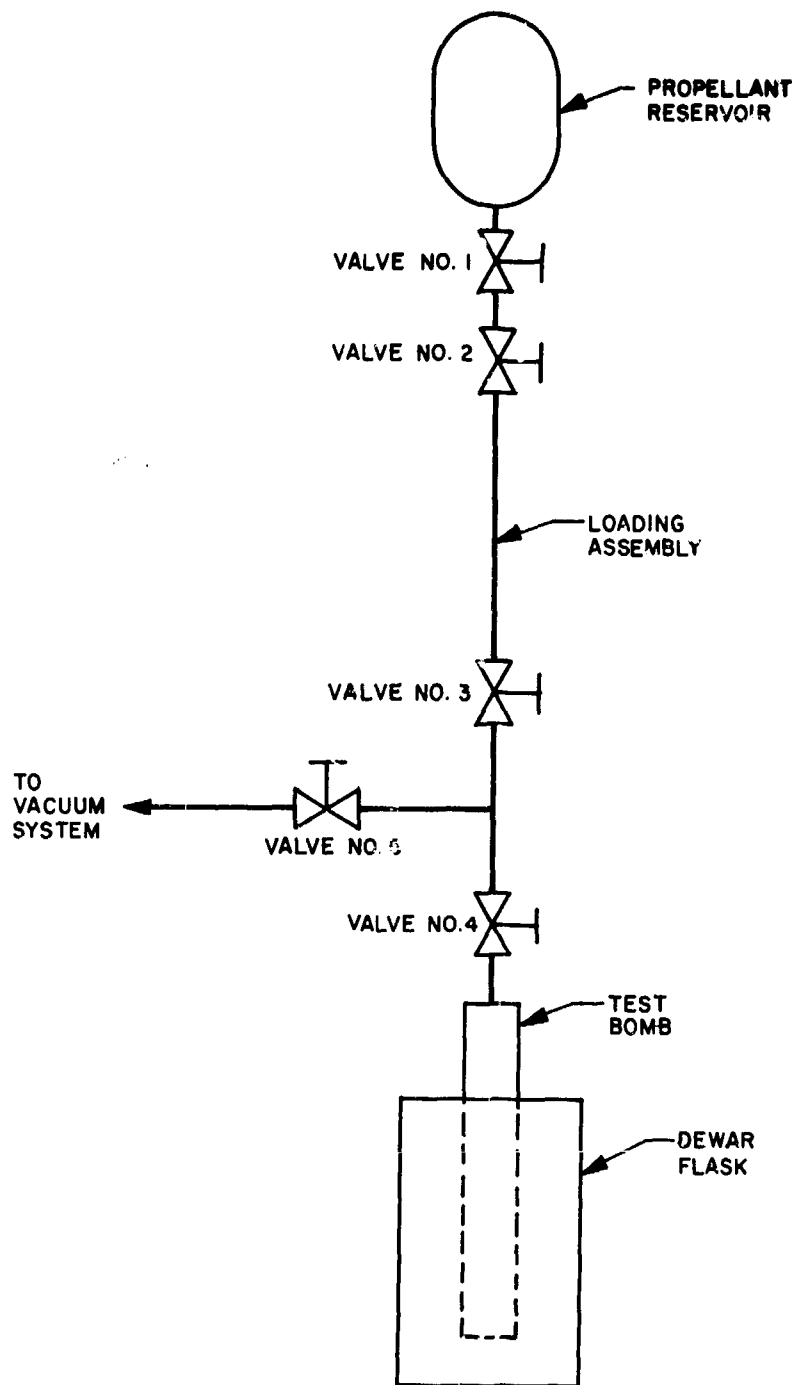


Figure 2. Loading Schematic

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Seven-day compatibility tests of the nonmetal specimens have been repeated with NT0 (military specification) and INT0-2, both immersed in the liquid and in contact with the vapor phase at ambient temperature.

The first three materials (Kel-F, Teflon, and Kynar) appeared unaffected by either NT0 or INT0-2 except for color change and a very slight surface softness. The Viton A exposed to both NT0 and INT0-2 exhibited extreme swelling (nearly twice the original size) as observed upon removal from the test bombs. After exposure to atmosphere for 30 minutes, the specimens reduced in size to approximately one and one-tenth the original size. There was some splitting around the inside diameter of the specimens exposed to the liquid phase of both the NT0 and INT0-2. The Viton A specimen exposed to liquid phase INT0-2 had approximately one-fourth the resiliency of the control specimen compared to approximately three-fourths for the specimen exposed to the liquid phase NT0. In addition, the Viton A specimen exposed to liquid phase INT0-2 was the only specimen which lost weight.

Although the Butyl rubber also exhibited extreme swelling as immediately observed upon removal from the bomb, the swelling was not as great as that of the Viton A and there was no cracking. After exposure to the atmosphere for 30 minutes, the specimens returned to their original size with no apparent color change.

Significantly, there was considerably less reaction between the nonmetals and INT0-2 than between the nonmetals and INT0-1. There was little reaction between INT0-2 and the Butyl rubber but the INT0-1 dissolved nearly all of the Butyl rubber. The amount of remaining inhibitor was also greater during the INT0-2 test. Pretest FNO_2 content was 3.0 mole percent (in the supply bomb) and posttest FNO_2 content was 1.5 mole percent (in the test bomb).

The 321 stainless-steel bomb used during the nonmetals test was prepassivated with an NT0 + FNO_2 solution containing 3.0 mole percent FNO_2 . The solution remained in the bomb for 24 hours at 23 C. All corrosion test bombs are stored with the valve end down for ease of acquisition of the analysis samples and to eliminate disturbance of the test specimens.

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All specimens were weighed after 72 hours and 260 hours exposure to atmosphere. The results, along with appropriate comments, are presented in Tables 5 and 6 and in Fig. 3 through 5. Figure 3 shows the test samples from INT0-1 and NT0, and Fig. 4 shows those from INT0-2 and NT0. Figure 5 shows the residue remaining after the INT0-2 and NT0 were allowed to evaporate.

A comparison of the results of the 30-day ambient test with those of the 20-month ambient test should provide the best measure of the effectiveness of FNO_2 in inhibiting corrosivity by NT0. It is to be expected that the 30-day coupons immersed and in contact with the vapor from FNO_2 + NT0 will show as great a change in weight and appearance as those exposed to wet NT0 because of the formation of a passivated protective layer. If the layer adequately protects the metals from corrosion, little or no additional change in weight or appearance should occur to the specimens exposed to the FNO_2 - NT0 over the 20-month period whereas the specimens exposed to wet NT0 would be expected to be further attacked.

Galvanic Corrosion

The conductances of wet NT0 (0.13 weight percent H_2O) and INT0-1 (5 weight percent F_3NO) prepared from this NT0 have been determined as $9 \times 10^{-12} \text{ ohm}^{-1} \text{ cm}^{-1}$ and $4 \times 10^{-12} \text{ ohm}^{-1} \text{ cm}^{-1}$ respectively. These two measurements complete the conductance studies on INT0-1 and its components. From the measured values of the conductances, which lie in the region of 10^{-11} to $10^{-12} \text{ ohm}^{-1} \text{ cm}^{-1}$, it is apparent that galvanic (bimetallic) corrosion will not be a serious problem in the use of INT0-1.

Because INT0-1 is being discarded in favor of INT0-2 it will be necessary to repeat the conductivity tests on INT0-2. These will be carried out during the next quarter.

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TABLE 5
SEVEN-DAY COMPATIBILITY TEST OF NONMETALS WITH NTO

Material	Weight Before, grams	Weight After, grams	Weight Change After 72 Hours, grams	Percent Weight* Change After 72 Hours	Percent Weight* Change After 240 Hours	Comments
Liquid Phase						
Kel-F	0.5235	0.5468	0.0233	4.4	3.5	Surface slightly softer in both solutions and both phases; color changed to light amber; no apparent size change
Kynar	0.4413	0.4578	0.0165	3.7	2.4	Color changed to light green; no apparent size change
Bu' 71 Rubber	0.3183	0.3428	0.0245	7.7	5.4	Extreme swelling to 1-3/4 times original size as observed immediately upon removal; surface slightly spongier than control specimen; no apparent color change
Teflon	0.5462	0.5480	0.0018	0.3	0.2	No apparent change in size or surface hardness; color changed to buff but returned to original color upon extended exposure to atmosphere (240 hours)
Viton A	0.5940	0.6341	0.0401	6.7	7.2	Extreme swelling to approximately twice original size observed upon removal from bomb; reduced to 1.1 original size in 30 minutes upon exposure to atmosphere; greater swelling, cracking, and warping in liquid phase than in vapor phase; slight reduction in elasticity
Vapor Phase						
Kel-F	0.5318	0.5554	0.0236	4.4	3.5	Color changed to light amber; surface slightly softened; no apparent size change
Kynar	0.4377	0.4542	0.0165	3.8	2.5	No apparent size change; color changed to light green; very slight surface softening
Bu' 71 Rubber	0.3383	0.3690	0.0307	9.1	6.6	Extreme swelling observed upon removal from bomb; no apparent color change; surface appeared spongier than control specimen
Teflon	0.5357	0.5373	0.0016	0.3	0.2	Color changed to buff but returned to normal upon extended exposure to atmosphere (240 hours)
Viton A	0.5740	0.6183	0.0443	7.7	8.5	Extreme swelling to about twice original size upon removal from bomb; returned to 1.1 original size after 30-minutes exposure to atmosphere; no apparent color change; slight reduction in elasticity

* Percent weight change based on original weight

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TABLE 6
SEVEN-DAY COMPATIBILITY TEST OF NONMETALS WITH NTO + FNO 2

Material	Weight Before, grams	Weight After, grams	Weight Change After 72 Hours, grams	Percent Weight Change After 72 Hours	Percent Weight Change After 240 Hours	Comments
Liquid Phase						
Kel-F	0.5322	0.5562	0.0240	4.5	3.6	Surface slightly softer in both solutions and in both phases after test; color after test, light amber; no apparent size change
Kynar	0.4336	0.4496	0.0160	3.7	2.4	Surface slightly softer but not as evident as Kel-F; color changed to light green; no apparent size change
Butyl Rubber	0.3077	0.3296	0.0219	7.1	4.8	Exhibits a slight sponginess compared to control specimens; characteristics similar in liquid and vapor phase; NTO and INT0-2 extreme swelling observed immediately upon removal (1-3/4 times original size)
Teflon	0.5484	0.5498	0.0014	0.3	0.07	No apparent change in surface hardness or size; color changed to buff; after extended exposure to atmosphere, color changed back to original
Viton A	0.5410	0.5731	-0.0079	-1.4	-2.5	Extreme swelling to nearly twice original size observed immediately upon removal from test bomb, reduced to 1.1 original size after 30-minutes exposure to atmosphere; slightly greater warping, cracking, and reduction in elasticity in INT0-2 than in NTO
Vapor Phase						
Kel-F	0.5564	0.5500	0.0233	4.5	3.6	Changed color to light amber; surface slightly softer than control specimen; no apparent change in size
Kynar	0.4342	0.4526	0.0164	3.8	2.4	Color changed to light green; no apparent change in size; very slight softening of surface
Butyl Rubber	0.3170	0.3424	0.0258	8.1	6.0	Extreme swelling observed immediately after removal from bomb; surface spongier than control specimen; no apparent change in color
Teflon	0.5490	0.5504	0.0016	0.3	0.1	No apparent change in surface softness or size; color changed to buff but returned to original color after extended exposure to atmosphere (240 hours)
Viton A	0.4094	0.4449	0.0407	6.7	6.2	Extreme swelling to nearly twice original size observed immediately upon removal from test bomb; reduced to 1.1 original size after 30-minutes exposure to atmosphere; reduction in elasticity as in liquid phase; less warping and cracking than in liquid phase; no apparent color change

*Percent weight change based on original weight

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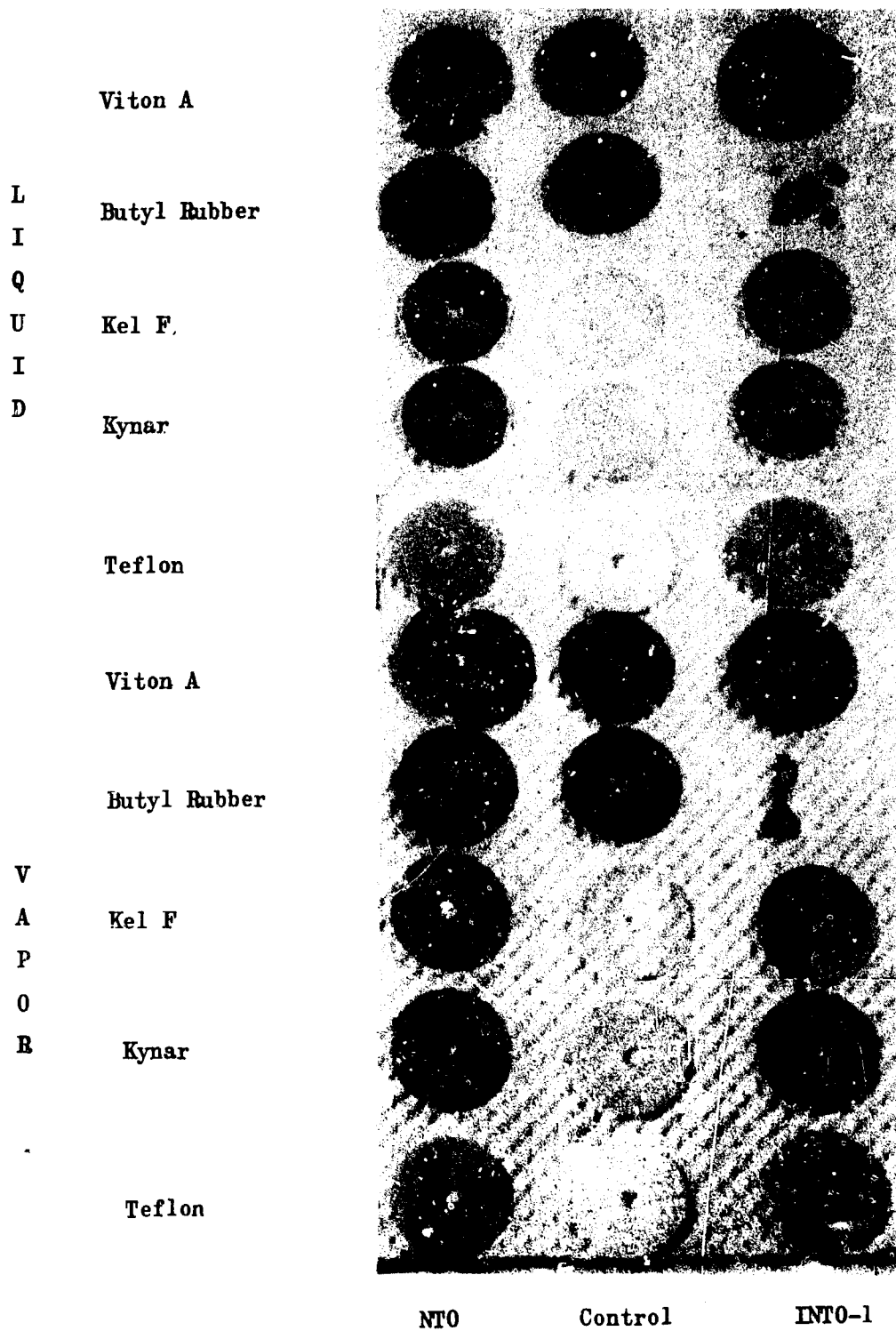
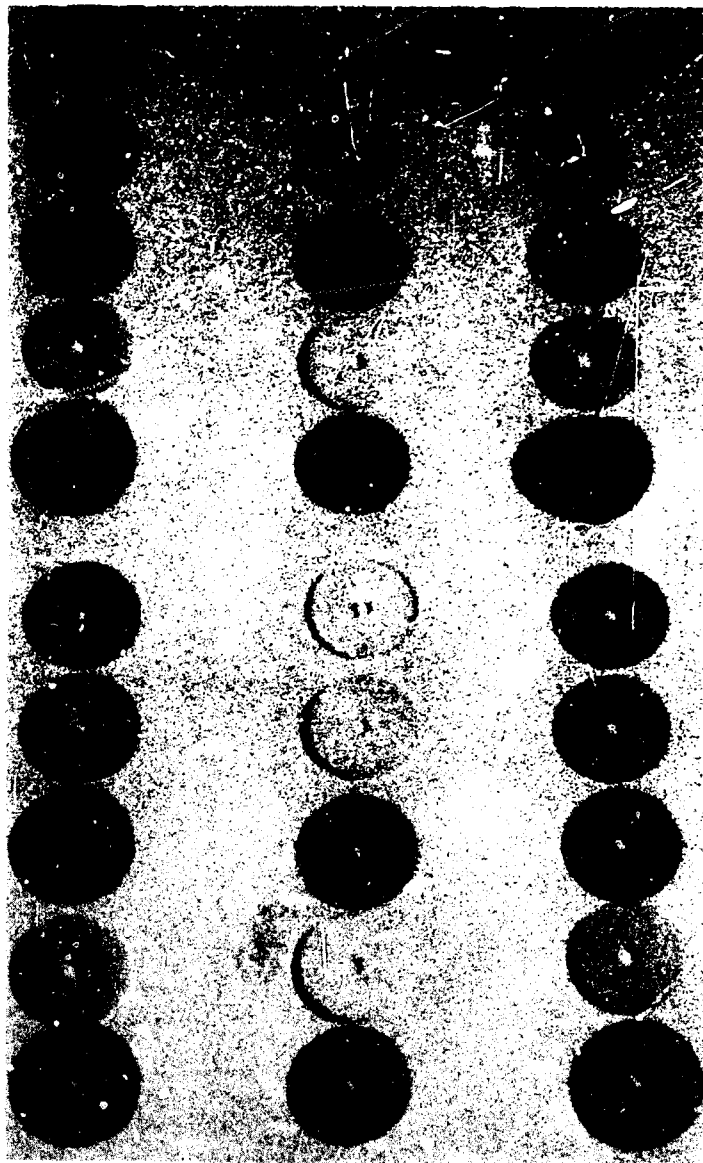


Figure 3. Seven-Day Test Nonmetals

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Kel-F
L Kynar
I
Q Butyl Rubber
U
I Teflon
D
Viton A
Kel-F
Kynar
V
A
P Butyl Rubber
O
R Teflon
Viton A



NTO

CONTROL

INT0-2

Figure 4 . Seven-Day Test Nonmetals

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INTO-2

Figure 5. Seven-Day Test Residue

NT0

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Storability and Compatibility

INTO-1 was tested for storability and constituents compatibility in 6061 aluminum, 1018 carbon steel, 321 stainless steel, and nickel containers at ambient temperature, 53 C and 70 C. The tests were carried out under two conditions of bomb prepassivation: (1) mild, using F_3NO at 70 C overnight, and (2) vigorous, using ClF_3 at 70 C overnight. The bombs had a capacity of 8 to 10 milliliters and each was loaded with 4 to 6 grams of a 4.9-percent F_3NO in NT0 mixture. The samples were analyzed by infrared spectrometry and gas chromatography both immediately after loading and after appropriate time periods. The data are summarized in Tables 7 through 9.

An examination of the analytical data immediately reveals that the components of INTO-1 are not compatible at elevated temperatures. As a result, emphasis on the program has been shifted exclusively to INTO-2 (FNO_2 + NT0).

Because INTO-2 was formed by the reaction of F_3NO with NT0, the compatibility tests originally started with INTO-1 were continued using the same solutions in the same bombs. The data from these tests are also contained in Tables 7 through 9. These results indicated that INTO-2 is storable at elevated temperatures, in sufficiently passivated containers, for at least 55 days (45 days at 53 C and 10 days at 70 C). These tests are being continued and the contents of the bombs will be periodically analyzed.

Vapor Pressure and Solubility

Equally important to determining the corrosivity of INTO on materials of construction is determining the solubility of FNO_2 (boiling point: -72.4 C) in NT0. Preliminary experiments have indicated that the solubility lies within operationally acceptable limits, but exact quantitative data are necessary to design an operational propellant.

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TABLE 7

STORABILITY OF INTO-1 AT AMBIENT TEMPERATURE

Material	Mole Percent AMOX at Start	Mole Percent AMOX After 34 Hours	Mole Percent AMOX After 80 Hours	Mole Percent AMOX After 160 Hours
Stainless Steel (Working Solenoid Bomb)	4.9	—	—	4.6
321 Stainless Steel	4.9	3.4	3.3	2.3
Carbon Steel-1018	3.6	1.6	1.0	1.0
Al-6061	3.9	3.4	3.2	2.8
Nickel	4.2	3.4	3.5	3.2

Passivation: AMOX at 1500 mm absolute pressure, STP, 24 hours at 70 C

Working Solution: 755 grams NTC plus nominal 5 mole percent AMOX in 1-liter stainless-steel high-pressure bomb

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TABLE 8

STORABILITY AT 53 C

Material	Passivation	Mole Percent F_2NO at Start	Mole Percent $FeNO_2$ at Start	Mole Percent F_2NO After 34 Hours	Mole Percent $FeNO_2$ After 34 Hours	Mole Percent F_2NO After 80 Hours	Mole Percent $FeNO_2$ After 80 Hours	Mole Percent F_2NO After 100 Hours	Mole Percent $FeNO_2$ After 100 Hours	Mole Percent F_2NO After 30 Days	Mole Percent $FeNO_2$ After 30 Days
321 Stainless Steel	1	4.0	0	0.4	1.3	0	0				
Carbon Steel-1018	1	4.1	0	0.3	1.1	0	0				
Al-6061	1	3.6	0	0.5	0	Trace	0				
Nickel	1	3.8	0	0.3	3.3	0	4.0				
321 Stainless Steel	2	2.3	0	0.8	4.0	0	4.2	0	4.2	0	4.3
Al-6061	2	2.7	0	0.7	2.9	0	4.7	0	4.7	0	4.8
Nickel	2	2.5	0	0.9	3.2	0	4.5	0	4.5	0	4.5
Nickel Body with Monel Valve	3	3.4	0	1.5	3.8	0.25	4.1				

- Passivation:
1. F_2NO , 1500 mm Hg absolute pressure, STP, 24 hours, 70 C
 2. Three of bombs used with passivation procedure 1 were further passivated with one atmosphere ClF_3 (STP) at 70 C for 24 hours
 3. Single bomb previously used for reaction of 4 grams 50/50 F_2NO - NO mixture at 70 C for 5 days

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TABLE 9
STORABILITY AT 70 C

Material	Passivation	Mole Percent F_3NO at Start	Mole Percent $FN O_2$ at Start	Mole Percent F_3NO After 34 Hours	Mole Percent $FN O_2$ After 34 Hours
321 Stainless Steel	1	4.0	0	0	0
Carbon Steel-1018	1	3.9	0	0	0
Al-6061	1	2.8	0	0	0
Nickel	1	4.1	0	0	2.5
321 Stainless Steel	2		4.3		After 10 Days 4.2
Al-6061	2		4.8		4.6
Nickel	2		4.5		4.5

Passivation: 1 F_3NO , 1500 mm Hg absolute pressure, STP, 24 hours, 70 C;

2 Three bombs previously passivated as in procedure 1, then further passivated with one atmosphere of ClF_3 (STP) at 70 C for 24 hours, and stored for 45 days at 53 C; temperature raised to 70 C and storage continued

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The solubility of FNO_2 in NTO will be determined by the familiar vapor pressure method. For calibration, it will be necessary to check the vapor pressure measurements over the range of 14 to 163 F (critical temperature) on a sample of pure FNO_2 . A precision, temperature-compensated Heise gage will be used for this work.

The vapor pressure and solubility studies proposed for INTO-1 in the previous quarterly report will now be carried out for INTO-2 taking into consideration the lesser volatility and higher critical temperature of FNO_2 .

Transfer Effects

Because INTO-2 is replacing INTO-1 in this program the investigation of the erosive effects of transfer on materials will be performed with INTO-2 according to the plans delineated in the first quarterly report.

Venting

The loss of FNO_2 from INTO-2 by venting will be determined as a function of time by the method previously proposed for the loss of F_3NO from INTO-1 .

RECLAMATION AND PREPARATION STUDIES

Because of the unique method of preparation of INTO-2 , the problems of reclamation of NTO and preparation of INTO must be considered together.

INTO-2 was first prepared by the reaction of F_3NO with NTO . This method of preparation is obviously impractical for large-scale production because of the very high cost of F_3NO . The method now used to prepare INTO-2 is the reaction of F_2 with NTO . This has been carried out by condensing F_2 into a stainless-steel bomb at -196°C and allowing the mixture to warm to room temperature. This method of preparation has been

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reported by Faloon and Kenna Ref. 1. Later in the program attempts will be made to make INTO-2 by adding F_2 to NTO at ambient temperature, probably by bubbling in the F_2 . If the latter method is successful, it should be possible to design a system for making INTO-2 by adding F_2 to either in-specification or out-of-specification NTO in field storage tanks.

The problem concerning the presence of out-of-specification NTO in storage tanks has persisted for some time. Sometimes contaminated storage tanks will stand for long period of time before their contents are discarded. The expense of either discarding or reprocessing wet NTO is prohibitive. It might be possible to solve this problem by converting the out-of-specification NTO to INTO by the addition of $FN O_2$. Hence, it is very worthwhile to examine the possibility of such a reclamation process.

The feasibility of upgrading NTO with as much as 0.5-percent water equivalent is to be studied during this program.

INTO-2, made both from F_3NO -NTO and F_2 -NTO, has been shown by Turner bulb military specification analysis performed in a specially constructed aluminum apparatus (Ref. 2) to drastically reduce the water content originally present in the NTO and also to destroy added water (Table 10). The infrared analysis has shown that a corresponding reduction in $FN O_2$ content also occurred.

These experiments indicated that reclamation of NTO with 0.3-percent water equivalent is feasible.

TABLE 10
EFFECT OF INTO-2 ON WATER EQUIVALENT

<u>Oxidizer</u>	<u>Water Equivalent, weight percent</u>
NTO	0.15
INTO-2 (from F_2 + NTO)	0.02
INTO-2 (from F_2 + NTO after adding 0.15 weight percent H_2O)	0.02

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UNUSUAL PHENOMENA OR HAZARDS

Storability-compatibility tests run indicated that INTO-1 was not storable at elevated temperatures. These studies were discussed in the storability section of this report.

These studies confirm the storability problem of F_3NO in NTO caused by reaction at elevated temperatures. However, the studies also suggest that FNO_2 is a better water scavenger than F_3NO and is compatible with NTO at elevated temperatures. Because FNO_2 is more readily synthesized (fluorine + NTO), than F_3NO , its usefulness as a replacement for F_3NO in inhibited nitrogen tetroxide (INTO) is very promising.

An explosion occurred in a sealed Pyrex u.r.r. sample tube containing heat NTO being prepared for water analysis. The explosion occurred just after the tube had been sealed and the NTO (originally frozen at $-196^\circ C$) began melting. Some black specks of material were noted in the NTO just prior to the explosion. No injury resulted. It is suspected that some valve packing material may have worked loose and reacted with the NTO.

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PHASE II: ANALYTICAL STUDIES

INFRARED ANALYSIS

On examination of the cylinder of F_3NO for minor impurities by IR analysis it was found that it contained some FNO_2 which was removed by passing the F_3NO through a $-80^\circ C$ AlF_3 trap. FNO_2 was also found to be removed by passing the F_3NO through a 6-foot stainless-steel tube packed with Ascarite.

The thermal stability of INTO-1 was mainly followed by IR analysis and the results are given elsewhere in this report. Because INTO-1 is being replaced by INTO-2 the development of an accurate analysis for FNO_2 was necessary. A calibration for FNO_2 has been obtained in the infrared region by using the intensity of the 12.18-micron band of FNO_2 as a quantitative measure of the FNO_2 content. The calibration curve is shown in Fig. 6. This method was used to show that the reaction of F_2 and NTO did produce FNO_2 in sufficient quantity to yield INTO-2. A spectrum of INTO-2 is shown in Fig. 7. Figure 8 shows the calibration curve for the 2.51-micron band of HF which is used to determine the HF which is formed in INTO-2.

NUCLEAR MAGNETIC RESONANCE ANALYSIS

The n.m.r. technique for water determination has been used to check the water equivalent of the NTO samples used during the nonmetals and metals corrosion compatibility tests and in the galvanic corrosion tests. The NTO used was found to contain less than 0.10 weight percent water equivalent. To prepare wet NTO it will be necessary to add extra water to the available NTO. F^{19} n.m.r. confirmed the formation of FNO_2 by the reaction between F_2 and NTO.

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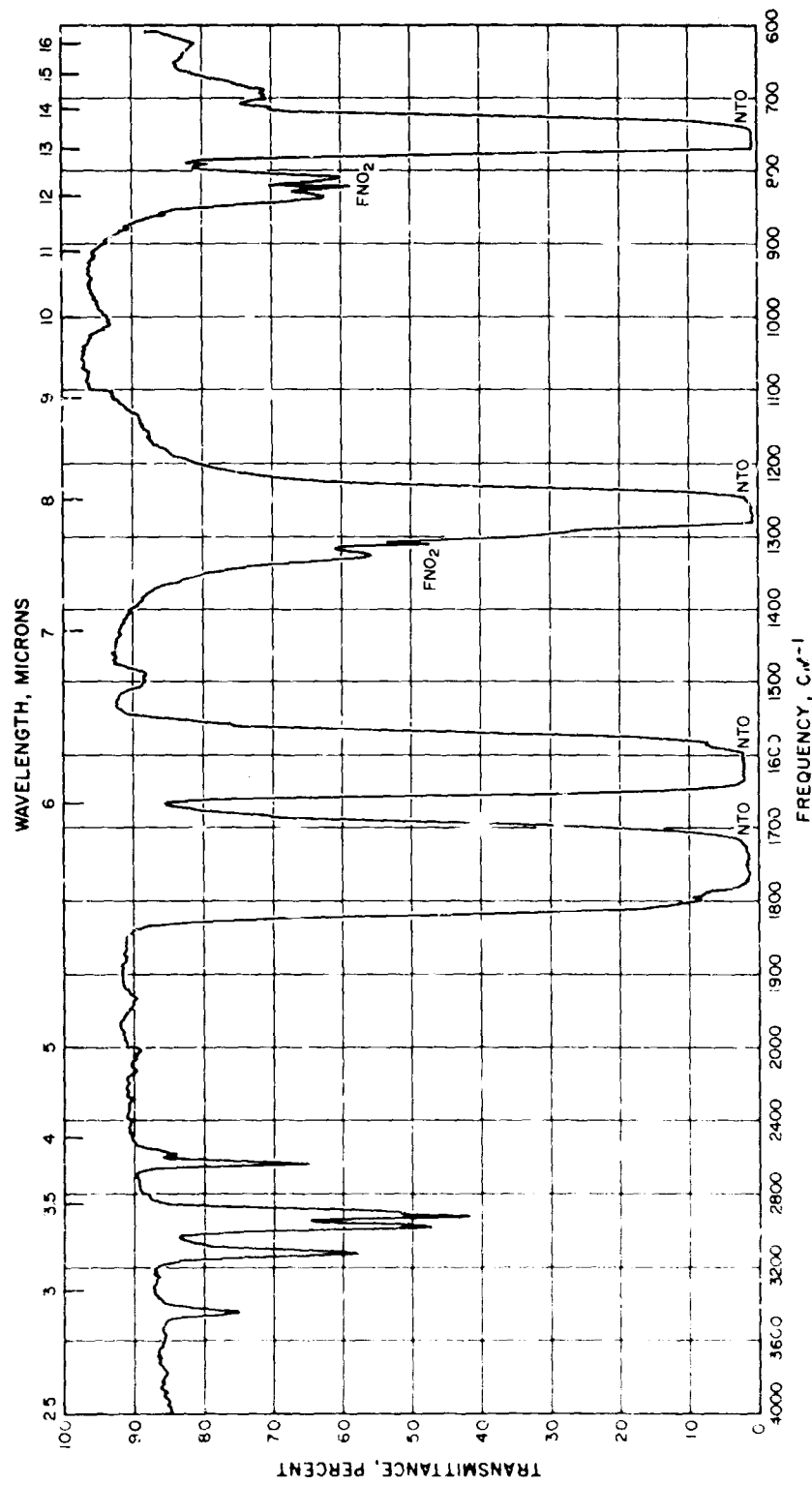


Figure 6. Infrared Calibration Curve

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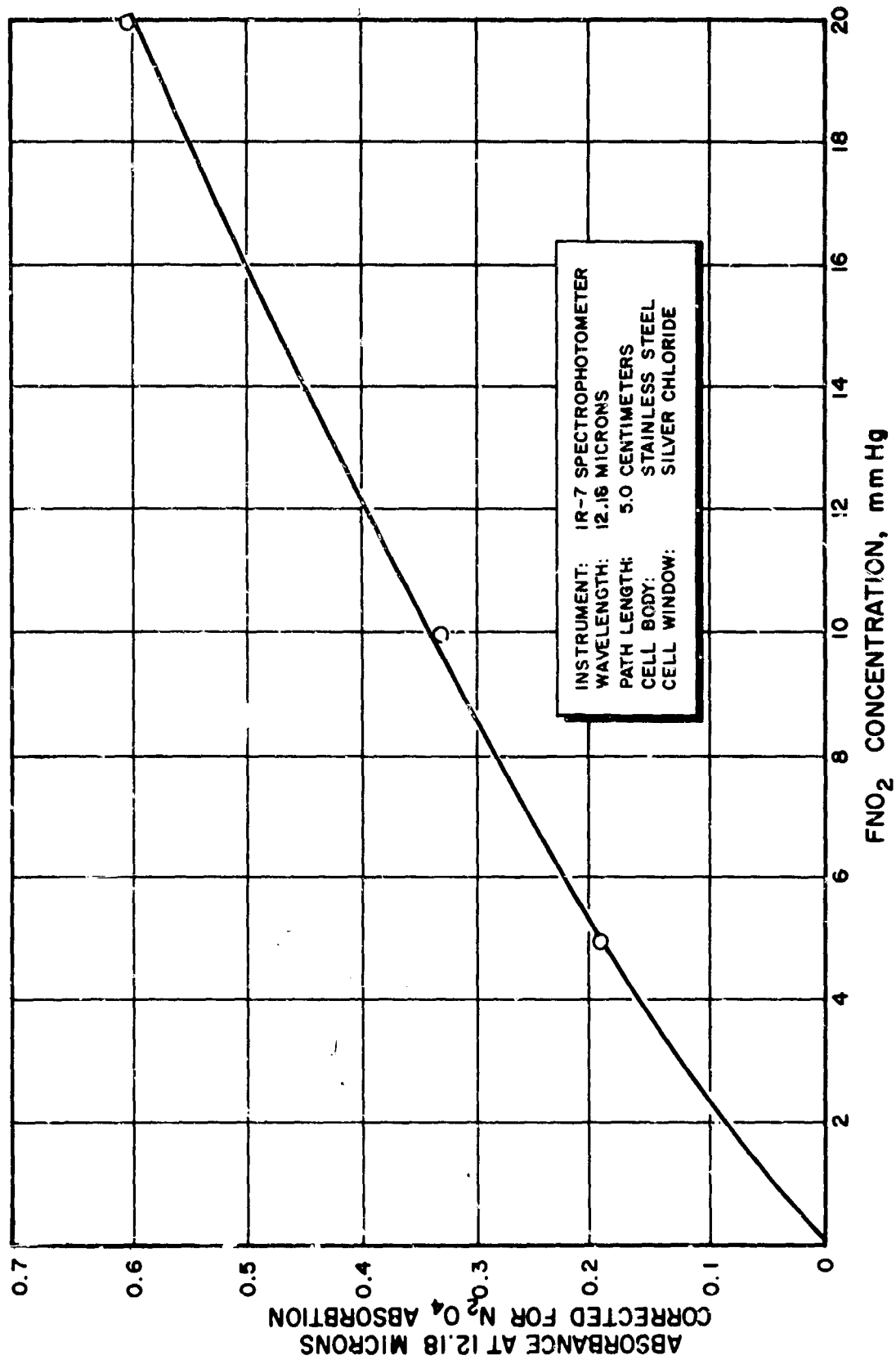


Figure 7. Spectrophotometer Calibration Curve for FNO_2 in N_2O_4

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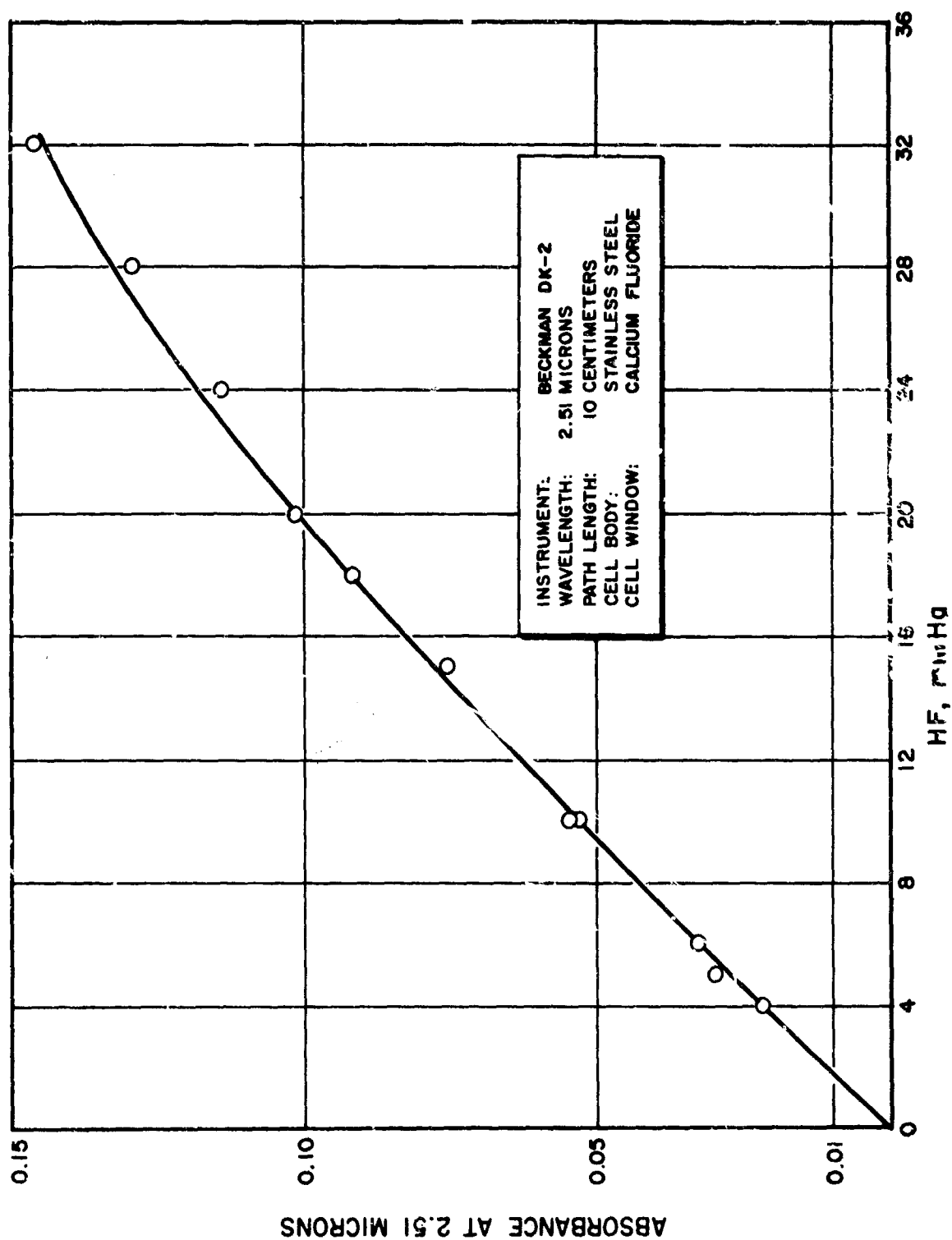


Figure 8. Spectrophotometer Calibration Curve for HF

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WATER EQUIVALENT TESTING

The all-aluminum model of the military specification water equivalency apparatus was used on a sample of NT0 tested by the n.m.r. method. Using the aluminum apparatus, the water equivalent was found to be 0.21 weight percent; using n.m.r., .13 weight percent; and using the glass military specification apparatus, 0.15 weight percent.

After using the aluminum apparatus for this determination, a considerable amount of sludge was found in the bulb. A sample of INT0-1 made from the above NT0 had a water equivalent of 0.04 and 0.05 weight percent as determined by the glass military specification apparatus. These latter results may be slightly high because of reaction of HF with the glass.

The aluminum apparatus was then passivated with 48-percent HF in H_2O . The NT0 analyzed using this apparatus was found to contain 0.15 weight percent water equivalent but this was reduced to 0.03 weight percent when the NT0 was converted to INT0-2 by the addition of F_2 . To this sample of INT0-2 water was added equivalent to 0.2 weight percent; the water equivalent was measured and found to still be 0.03 weight percent. No evidence was found of the sludge which had been produced by reaction of NT0 with the same apparatus prior to passivation with HF.

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FUTURE EFFORT

The galvanic corrosion studies will be repeated for INTO-2. The loading of samplers for short- and long-term corrosivity tests will be completed and the tests will be continued. Work will proceed on the vapor pressure, solubility, and freezing point studies. Reclamation studies will be carried out in conjunction with improved practical methods of preparing INTO-2 from fluorine and NT0. Analytical methods will be improved or modified as required to support the program.

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1. Falcon, A. V. and W. B. Kenna, J. Am. Chem. Soc., 73, 2937 (1951).
2. R-6354-1, Inhibited N₂O₄, First Quarterly Progress Report, Rocketdyne, a Division of North American Aviation, Inc., Canoga Park, California, October 1965. CONFIDENTIAL

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13. ABSTRACT This program was originally concerned with evaluating a new storable liquid oxidizer, INTO, which is NTO inhibited with a fluorine-containing oxidizer, F_3NO . Storability results have shown that F_3NO is not compatible with NTO at elevated temperatures. FNO_2 has been substituted as the inhibiting fluorine oxidizer and this new propellant system is called INTO-2. Its constituents have been found to be compatible at 70 C. Long- and short-term corrosivity tests have been initiated. The 7-day compatibility tests with nonmetallics have been completed in NTO, INTO-1, and INTO-2. Results indicate that INTO-2 is more compatible than INTO-1 with the materials studied. There was little difference between NTO and INTO-2. Conductance measurements were completed on INTO-1. Results indicated that galvanic corrosion will not be a problem. INTO-2 has been prepared by reaction of fluorine with NTO and the resulting mixture has been shown to react with added water to bring the water equivalent down to 0.03 percent. Analytical methods have been developed for the analysis of INTO-1 and INTO-2. (C)		

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14 KEY WORDS	LINK A		LINK B		LINK C	
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